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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the Application of:

Wang et al.

Serial No.: 10/687,471

Filed: October 15, 2003

For: COLD-WATER SOLUBLE EXTRUDED
STARCH PRODUCT

Atty. Docket No.: 006401.00417

Group Art Unit: unassigned

Examiner: unassigned

**PETITION UNDER 37 C.F.R. 1.181 TO HAVE THE NOTICE OF INCOMPLETE
NONPROVISIONAL APPLICATION WITHDRAWN**

Mail Stop Petitions
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

This Petition is being filed in response to the Notice of Incomplete Nonprovisional Application mailed January 26, 2004. A petition fee (per 37 CFR 1.17(h)) accompanies this Petition. If the Office should decide that Applicant is entitled to a refund of the petition fee, Applicant respectfully requests that any such refunds be deposited to Deposit Account No. 19-0733.

Applicant respectfully requests that the Notice of Incomplete Nonprovisional Application be withdrawn, Patent Application No. 10/687,471 be assigned a filing date of October 15, 2003, and the application be allowed to continue to substantive examination. The Notice of Incomplete Nonprovisional Application was issued because the application was filed without drawings (37

CFR 1.53(b)). Applicant inadvertently omitted five Drawing Sheets from Parent Patent

03/24/2004 AWONDAF1 00000083 190733 10687471

01 FC:1460 130.00 DA

Appln No.: 10/687,471
Response Dated: March 18, 2004
Reply to Office Action of: January 26, 2004

Application No. 09/863,928 when filing this divisional application on October 15, 2003. This patent application was clearly designated as a divisional application of said parent application. The entire contents of the parent patent application were also expressly incorporated by reference in the instant application.

The attached Paper entitled "AMENDMENT IN RESPONSE TO NOTICE OF INCOMPLETE NONPROVISIONAL APPLICATION DATED JANUARY 26, 2004" is being submitted to the Office to include the omitted drawings from the parent patent application.

Therefore, Applicant respectfully requests that the attached Amendment be entered, the Notice of Incomplete Nonprovisional Application be withdrawn, Patent Application No. 10/687,471 be assigned a filing date of October 15, 2003, and the application be allowed to continue to substantive examination. The Office is invited to contact the undersigned at the telephone number listed below.

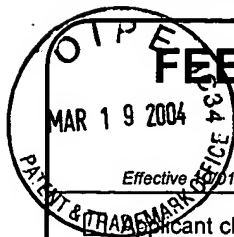
Respectfully submitted,

BANNER & WITCOFF, LTD.

By: 
Aseet Patel (Reg. No. 53,874)

Ten South Wacker Drive
Suite 3000
Chicago, Illinois 60606-7407
(312) 463-5000

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FEE TRANSMITTAL for FY 2004

Effective 10/1/2003. Patent fees are subject to annual revision.

Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT (\$) 130

Complete if Known

Application Number 10/687,471
 Filing Date October 15, 2003
 First Named Inventor Wang et al.
 Examiner Name (TBA)
 Art Unit (TBA)
 Attorney Docket No. 06401.00417

METHOD OF PAYMENT (check all that apply)

☐ Check ☐ Credit card ☐ Money ☐ Other ☐ None
 Order
☒ Deposit Account:

Deposit Account Number 19-0733

Deposit Account Name Banner & Witcoff, LTD.

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FEE CALCULATION

1. BASIC FILING FEE

Large Entity		Small Entity		Fee Description	Fee Paid
Fee Code	Fee (\$)	Fee Code	Fee (\$)		
1001	770	2001	385	Utility filing fee	
1002	340	2002	170	Design filing fee	
1003	530	2003	265	Plant filing fee	
1004	770	2004	385	Reissue filing fee	
1005	160	2005	80	Provisional filing fee	
SUBTOTAL (1)					(\$) 0

2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE

Total Claims	Extra Claims	Fee from below	Fee Paid
Independent Claims			
Multiple Dependent			

Large Entity		Small Entity		Fee Description
Fee Code	Fee (\$)	Fee Code	Fee (\$)	
1202	18	2202	9	Claims in excess of 20
1201	86	2201	43	Independent claims in excess of 3
1203	290	2203	145	Multiple dependent claim, if not paid
1204	86	2204	43	** Reissue independent claims over original patent
1205	18	2205	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) (\$) 0

**or number previously paid, if greater; For Reissues, see above

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity		Small Entity		Fee Description	Fee Paid
Fee Code	Fee (\$)	Fee Code	Fee (\$)		
1051	130	2051	65	Surcharge - late filing fee or oath	
1052	50	2052	25	Surcharge - late provisional filing fee or cover sheet.	
1053	130	1053	130	Non-English specification	
1812	2,520	1812	2,520	For filing a request for reexamination	
1804	920*	1804	920*	Requesting publication of SIR prior to Examiner action	
1805	1,840*	1805	1,840*	Requesting publication of SIR after Examiner action	
1251	110	2251	55	Extension for reply within first month	
1252	420	2252	210	Extension for reply within second month	
1253	950	2253	475	Extension for reply within third month	
1254	1,480	2254	740	Extension for reply within fourth month	
1255	2,010	2255	1,005	Extension for reply within fifth month	
1401	330	2401	165	Notice of Appeal	
1402	330	2402	165	Filing a brief in support of an appeal	
1403	290	2403	145	Request for oral hearing	
1451	1,510	1451	1,510	Petition to institute a public use proceeding	
1452	110	2452	55	Petition to revive - unavoidable	
1453	1,330	2453	665	Petition to revive - unintentional	
1501	1,330	2501	665	Utility issue fee (or reissue)	
1502	480	2502	240	Design issue fee	
1503	640	2503	320	Plant issue fee	
1460	130	1460	130	Petitions to the Commissioner	130
1807	50	1807	50	Processing fee under 37 CFR 1.17 (q)	
1806	180	1806	180	Submission of Information Disclosure Stmt	
8021	40	8021	40	Recording each patent assignment per property (times number of properties)	
1809	770	2809	385	Filing a submission after final rejection (37 CFR § 1.129(a))	
1810	770	2810	385	For each additional invention to be examined (37 CFR § 1.129(b))	
1801	770	2801	385	Request for Continued Examination (RCE)	
1802	900	1802	900	Request for expedited examination of a design application	

Other fee (specify) _____

*Reduced by Basic Filing Fee Paid

SUBTOTAL (3) (\$) 130

SUBMITTED BY

Complete (if applicable)

Name (Print/Type) Aseet Patel Registration No. (Attorney/Agent) 53,874 Telephone (312) 463-5000
 Signature *Aseet Patel* Date March 19, 2004

WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

This collection of information is required by 37 CFR 1.17 and 1.27. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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	2A	2B	2C	2D	2E	2F	2G
Raw Material Information							
Substrate	B760	B760	B760	B760	B760	B760	B760
Dry Recipe % moisture	10-11	10-11	10-11	10-11	10-11	10-11	10-11
Feed Starch Rate (lbs/hr)	350	350	250	270	290	310	330
Cylinder Information							
Steam Flow to Cylinder lb/hr	12	12	12	12	12	12	12
Water Flow to Cylinder lb/hr	8	8	8	8	8	8	8
Extrusion Information							
Extruder Shaft Speed rpm	350	350	250	270	290	310	330
Extruder Motor Load %	51	51	35	40	41	46	47
Steam Flow to Extruder lb/hr	0	0	0	0	0	0	0
Water Flow to Extruder lb/hr	10	10	10	10	10	10	10
1 st Head Temp	98° F	97° F	96° F	96° F	95° F	95° F	97° F
2 nd Head Temp	135° F	136° F	124° F	125° F	125° F	126° F	126° F
3 rd Head Temp	144° F	198° F	129° F	131° F	132° F	135° F	136° F
4 th Head Temp	179° F	180° F	176° F	175° F	175° F	180° F	180° F
5 th Head Temp	292° F	295° F	231° F	233° F	233° F	236° F	239° F
Specific Mechanical Energy kW/ton	79	79	76	81	77	81	78

The starches produced were substantially completely soluble (over 99%).

Example 3

- The starch used in Example 1 was extruded on a Wenger TX57 Twin Screw
- 5 Extruder according to the following conditions and with the screw configuration shown in Figure 5.

Raw Material Information	
Substrate	B790
Dry Recipe % moisture	~11%
Dry Recipe Rate lb/hr	175
Feed Screw Speed rpm	15
Cylinder Information	
Cylinder Speed rpm	278
Steam Flow to Cylinder lb/hr	0
Water Flow to Cylinder lb/hr	0
Extrusion Information	
Extruder Shaft Speed rpm	398
Extruder Motor Load %	42
Steam Flow to Extruder lb/hr	0
Water Flow to Extruder lb/hr	11
Knife Speed rpm	459
No. of Knives	2
1 st Head Temp	77° F
2 nd Head Temp	78° F
3 rd Head Temp	108° F
4 th Head Temp	133° F
5 th Head Temp	270° F
6 th Head Temp	
Die Hole Size & How many?	3mm/15
Die Pressure psi	500
Vacuum on/off inches of vac?	OFF
Specific Mechanical Energy kW/ton	148

The expanded, friable product thus formed needed no drying. The product was ground on a Wiley Mill followed by an Alpine Mill to give a powder.

The powder was mixed into water at room temperature. A paste was formed, thus evidencing the gelatinized nature of the product. The paste was drawn into a thin film using a Meyer Road and then left to dry overnight at 50% relative humidity and 72° F to form a clear, transparent film.

Example 4

Cold Water Soluble Hydroxypropyl Starch

A cross-linked hydroxypropyl starch (B992 PURE-GEL[®], available from Grain Processing Corporation of Muscatine, Iowa), and having a moisture content of about 11% was extruded on a Wenger TX52 Twin Screw Extruder according to the conditions provided below and using the screw configuration shown in Figure 6.

	4A	4B	4C
Raw Material Information			
Substrate	B992	B992	B992
Dry Recipe % moisture	~11	~11	~11
Feed Screw Rate rpm	12	12	12
Cylinder Information			
Cylinder Speed rpm	110	110	110
Steam Flow to Cylinder lb/hr	0	0	0
Water Flow to Cylinder lb/hr	27.1	27.1	27.1
Extrusion Information			
Extruder Shaft Speed rpm	160	160	160
Extruder Motor Load %	29	28	17
Steam Flow to Extruder lb/hr	0	0	6.4
Water Flow to Extruder lb/hr	5.5	16.7	4.8
1 st Head Temp			
2 nd Head Temp	32° C	33° C	42° C
3 rd Head Temp	32° C	33° C	42° C
4 th Head Temp	90° C	90° C	90° C
5 th Head Temp	90° C	90° C	90° C
6 th Head Temp	65° C	65° C	65° C
7 th Head Temp	62° C	57° C	65° C
8 th Head Temp	62° C	57° C	65° C
9 th Head Temp	63° C	63° C	65° C
Die Pressure kPa	1720	70	2760

The extruded product, which was in the form of a condensed bead was dried on a moving grate dryer and then ground into a powder.

Each powder was mixed into water at room temperature to give pastes at 12% solids (thus evidencing the gelatinized nature of the extruded product). The pastes were
5 evaluated for gel strength and clarity. Gel strength was determined using a Texture Analyzer, Stevens LFRA Texture Analyzer TA 1000, 1 cm diameter probe after one day refrigeration at 40° F. Clarity was determined by observation on a scale of 0 to 9, 0 being opaque and 9 being clearest. The following results were obtained.

Evaluation Test	Water Temperature	4A	4B	4C	B992 Starch (control)
Gel Strength	30° C	26	25	26	*
	40° C	24	49	51	*
	50° C	50	56	60	48
	65° C	49	60	62	103
Clarity	30° C	1	1	1	*
	40° C	3	5	4	*
	50° C	7	7	6	0
	65° C	9	8	8	8

*The control, B992 Starch, was not amenable to testing at 30° C and 40° C.

Starch B992 was not amenable to testing at 30° and 40° because these temperatures were too low to allow this starch to gelatinize. Gel strength reflects the thickening power of a product when the product is mixed with water (generally, a higher gel strength is preferred in many applications).

5

Example 5

Cold Water Soluble Hydroxyethyl Starch

A hydroxyethyl starch (K95F COATMASTER® starch available from Grain Processing Corporation of Muscatine, Iowa) and having a moisture content of about 10 11% was extruded on a Wenger TX57 Twin Screw Extruder under the following conditions and having the screw configuration shown in Figure 7.

Raw Material Information	
Substrate	K95F
Dry Recipe % moisture	~11%
Dry Recipe Rate lb/hr	146
Feed Screw Speed rpm	8
Cylinder Information	
Cylinder Speed rpm	3498
Steam Flow to Cylinder lb/hr	0
Water Flow to Cylinder lb/hr	0
Extrusion Information	
Extruder Shaft Speed rpm	324
Extruder Motor Load %	32
Steam Flow to Extruder lb/hr	0
Water Flow to Extruder lb/hr	13
Knife Speed rpm	1087
No. of Knives	1
1 st Head Temp	85° F
2 nd Head Temp	169° F
3 rd Head Temp	175° F
4 th Head Temp	203° F
5 th Head Temp	245° F
6 th Head Temp	
Die Hole Size & How many?	3/36
Die Pressure psi	200
Vacuum on/off inches of vac?	OFF
Specific Mechanical Energy kW/ton	110

The product was made into an aqueous paste containing 35% extrudate at room temperature (thus evidencing the gelatinized nature of the product). The paste was

tested in a Rapid Visco-Analyzer (Newport Scientific) by monitoring the rotational viscosity and was found to have a viscosity of 1800 cP at 50° C compared to a baseline viscosity measurement of the raw starting material of about 100cP, thus evidencing the gelatinized nature of the extruded product. Additionally, when the paste was tested in a
5 Rapid Visco-Analyzer by monitoring the rotational viscosity during a controlled heating of the paste, the product exhibited no gelatinization peak. The starting material exhibited a crisp, characteristic peak at 70° C.

Example 6

10 Tack Coating and Cooked Products

The expanded starch from Example 1, 18 parts by weight, was blended with MALTRIN® M180 (a maltodextrin available from Grain Processing Corporation of Muscatine, Iowa), 9 parts by weight to form a dry blend. Water, 73 parts by weight, was added to a kettle and stirred with a powered mixer so as to create a vortex. The dry
15 blend was slowly added to the vortex, and the contents were mixed for an additional 10 to 30 minutes to form an instant tack coating.

The tack coating may be applied to a dry feed product substrate, such as a corn curl, pretzels, snack mix, or like item. The product may be applied by spraying or ladling at a level of from about 1% to about 15% weight gain, including moisture.
20 Seasonings, including savory seasonings such as Cajun, barbeque, cheese, mustard, ranch, Creole, and the like, or sweet seasonings such as sugar and pareils, may be added, and may be applied in any suitable manner, such as by hand or using a seasoning applicator. The resulting coated product preferably is dried in an oven at a temperature ranging from 300° F to 450° F to a moisture content of from about 3% to 5%.

25

Example 7

Oil-Based Instant Coating

Soybean oil, 50 parts by weight, was added to a vessel equipped with good agitation. The cold water soluble starch from Example 1, 7 parts by weight, was added
30 to the stirred oil and mixing was continued in order to achieve a smooth mixture.

Water, 42 parts by weight, and lecithin, 1 part by weight, were added as an emulsifier and mixing was continued for 10 to 15 minutes in order to achieve a smooth mixture.

The coated product may be applied to a food substrate as discussed in Example 6. Preferably, the coated product is dried in an oven at 300° F to 350° F with forced air
5 to a moisture content of from 3% to 5% in the finished product.

Example 8

Coated Peanut Products

A dusting mixture was prepared by dry-blending together the product of
10 Example 1, 50 parts by weight, and MALTRIN® M100 (a maltodextrin available from Grain Processing Corporation, Muscatine, Iowa), 50 parts by weight. Blanched, unroasted medium runner peanuts were placed in a 16" ribbed candy pan rotating at 20 to 25 RPM. A 50% sucrose solution was poured into the pan in an amount effective to just wet the nuts to give about a 2% weight gain. The dusting mixture was
15 then applied until the surfaces of the dusted nuts appeared dry, to thus give about a 5 to 6% weight gain. The dusted nuts were then tumbled an additional 2 to 3 minutes, during which time they wet back. An additional dusting with the dusting mixture was administered in order to achieve a dry appearance. The dry appearing, dusted nuts were then recoated with the sucrose solution, and the resulting rewetted nuts were
20 dusted to dryness again with the dusting mixture. This alternating procedure of wetting with the sucrose solution followed by dusting to dryness with the dusting mixture was repeated until a final dry appearing dusted nut resulted having a 75 to 100% weight gain as compared to the starting peanuts. The coated nuts were roasted in an oven at 300° F for 40 minutes with occasional stirring to assure uniformity of the
25 roast. The roasted coated nuts were cooled to room temperature and placed back into the ribbed pan rotating at 20 to 25 rpm. Subsequently, the instant tack coating formulation from Example 6 was sprayed onto the roasted coated nuts to provide approximately 0.5% weight gain in a rotating pan in order to create a slight tackiness. McCormick Barbecue Seasoning F76161, 6% to 8% weight gain was added, and the
30 coated nuts were tumbled until the seasoning was well distributed.

The resulting coated product was dried in an oven to a moisture level of from 3% to 5%.

Example 9

5

Trail Mix Coating and Product

A mixture was prepared by dry-blending together sugar, 25 parts by weight, the product of Example 1, 15 parts by weight, MALTRIN QD[®] M500 (a maltodextrin available from Grain Processing Corporation, Muscatine, Iowa), 5 parts by weight, and lecithin, 0.2 parts by weight. Water, 54.8 parts by weight, was added to a kettle and stirred with a powered mixer so as to create a vortex. The dry blend was slowly added into the water at the top edge of the vortex, and the contents were mixed for an additional 10 minutes to form an instant trail mix coating.

The resulting coating was sprayed onto a commercially purchased trail mix, by a spray gun system in a tumbler at a level of 5% to 15% weight gain. The resulting coated trail mix was dried in an oven at 150° F to a moisture content of 10 to 12%.

Example 10

Tablet Coating

A coating for a 3/8" round lactose/micro-crystalline cellulose placebo tablet was made. The coating had the following composition.

Formulation

Ingredients	Percentage by Weight
Product from Example 1	12.0%
Water	88.0%
	100.0

To prepare the coating, the starch was mixed into water with good agitation. A Vector HiCoater HC 100 coating pan with 2 spraying guns was used to apply the

coating onto the tablets to result in a 2% weight gain on the tablet. The coating pan was set at the following conditions.

Inlet temperature	60-65°C
Exhaust temperature	38-42°C
Pan speed	8 RPM
Process air flow	590 CFM
Spray air volume	125 atomize/50 pattern PSI
Spray rate	130-150 ml/min.

5 A plasticizer, such as glycerin, polyethylene glycols (PEG), propylene glycol (PG), oleic acid, triacetin, and the like can be used to improve the physical and mechanical properties of starch. Surfactants such as di-glycerides, tartaric acid esters of fatty acids, propyleneglyco mono and diesters of fatty acids, polysorbate 60, calcium or sodium stearyl-2-lactylate, lactic stearate, sodium stearyl fumarate, 10 succinylated monoglyceride, ethoxylated mono and diglycerides, and the like optionally may be used to provide hydrophilicity. Likewise, polymers of gums, cellulose derivatives, starch derivatives or hydrolysis products, and microorganism products, synthetic polymers such as polyvinyl alcohol, polyvinyl acetate, polyurethane, polystyrene, and polyvinylpyrrolidone, and so forth can be used to 15 improve the performance of the starch, for example, by increasing the flexibility and strength of the film coating.

Example 11

Film Coating

20 A coating for a 3/8" round lactose/micro-crystalline cellulose placebo was made. The coating had the following composition.

Formulation

Ingredients	Percentage by Weight
Product of Example 1	5.0%
Hydroxypropyl methyl cellulose	5.0%
Propylene glycol	1.0%
Polysorbate 80	0.5%
PURE-DENT [®] B815 corn starch NF*	0.5%
Titanium Dioxide	2.0%
Color	0.2%
Water	<u>85.8%</u>
	100.0

*Available from Grain Processing Corporation, Muscatine, Iowa.

To prepare the coating, the starch was mixed into water with good agitation. A Vector HiCoater HC 100 coating pan with 2 spraying guns was used to apply the
5 starch to tablets to result in a 3% weight gain on the tablets. The coating pan was set at the following conditions:

Inlet temperature	65-70°C
Exhaust temperature	40-45°C
Pan speed	8 RPM
Process air flow	575-595 CFM
Spray air volume	125 atomize/50 pattern PSI
Spray rate	170-180 ml/min.

Example 12

10 The properties of the starch extrusion may be characterized in part by an Extruder Solubilization Point Value (ESPV), which may be calculated as follows.

$$\text{ESPV} = \frac{1.71 \times 10^6 \times (M + M_{ws}) \times D^4}{(T_h - T_l) (M (F_{ws} C_{ps} + F_{ww} C_{pw}) + M_{ws} C_{pw}) (F_{fww}^5 \times M \times A_{fg})}$$

wherein

M = mass flow rate of starch through extruder (kg/s)
 M_{ws} = flow rate of water through extruder (kg/s)
 D = diameter of extruder barrel (m)
 T_h = highest head temperature in barrel (° C)
 T_l = lowest head temperature in barrel (° C)
 F_{ws} = weight fraction of starch in feed
 F_{ww} = weight fraction of water in feed
 F_{fww} = weight fraction of water in the barrel
 A_{fg} = grams of starch from viscosity test*(g)
 C_{ps} = specific heat capacity of starch (J/kg)
 C_{pw} = specific heat capacity of water (4186 J/kg)

5 *From the method disclosed in "The Estimation of Starch Paste Fluidities."
 W.R. Fetzer and L.C. Kirst, J. Cereal Chem., American Ass'n of Cereal Chemists,
 Vol. 36, No. 2 (U.S., March, 1959).

10 Preferably, the ESPV is greater than or equal to 1.0. Following is a table of extrusion
 conditions and ESPVs for the extruded starch of Examples 1, 2 and 3.

Inputs	Ex. 3	Ex. 1A	Ex. 1B	Ex. 1C	Ex. 1D	Ex. 1E	Ex. 1F	Ex. 1G
SME, kW/ton	148	100	106.2	103.4	100.7	102	99.3	98.2
M, lb/hr	175	4200	4275	4300	4400	4400	4500	4500
Mws, lb/hr	11	296	298	303	303	307	325	319
T _b , deg F	270	320	287	312	313	319	372	306
T _i , deg F	77	101	144	95	127	95	110	97
Fws	0.89	0.884	0.884	0.884	0.884	0.887	0.887	0.887
Fww	0.11	0.116	0.116	0.116	0.116	0.113	0.113	0.113
Ffww	0.163	0.174	0.174	0.174	0.173	0.171	0.173	0.172
Dia, mm	57	144	144	144	144	144	144	144
Afg	36	36	36	36	36	36	36	36
M/SME	1.18	42.00	40.25	41.59	43.69	43.14	45.32	45.62
ESPV	1.0	1.1	1.7	1.1	1.3	1.1	0.9	1.2

Inputs	Ex. 2A	Ex. 2B	Ex. 2C	Ex. 2D	Ex. 2E	Ex. 2F	Ex. 2G
SME, kW/lb	79	79	76	81	77	81	78
M, lbs/hr	350	350	250	270	290	310	330
Mws, lbs/hr	30	30	30	30	30	30	30
T _b , deg F	292	295	231	233	233	236	239
T _i , deg F	98	97	96	96	95	95	97
Fws	0.895	0.895	0.895	0.895	0.895	0.895	0.895
Fww	0.105	0.105	0.105	0.105	0.105	0.105	0.105
Ffww	0.176	0.176	0.201	0.195	0.189	0.184	0.180
Dia, mm	57	57	57	57	57	57	57
Afg	13	13	13	13	13	13	13
M/SME	4.43	4.43	3.29	3.33	3.77	3.83	4.23
ESPV	1.0	1.0	1.1	1.1	1.2	1.2	1.3

All of the ESPVs were above 1.0.

Comparative Example

Unacceptably sticky products were prepared by extruding B790 PURE-COTE[®] starch on a Wenger TX144 Twin Screw Extruder under the following conditions.

5

Inputs	C-1A	C-1B	C-1C	C-1D	C-1E
SME, kW/ton	86.9	76.4	103.6	140	150
M, lbs/hr	3800	4100	4100	3000	3000
Mws, lbs/hr	380	370	382	238	300
T _h , deg F	269	269	269	293	305
T _l , deg F	97	87	90	96	86
Fws	0.88	0.884	0.884	0.88	0.884
Fww	0.12	0.116	0.116	0.12	0.166
Ffww	0.200	0.189	0.191	0.185	0.196
Dia, mm	144	144	144	144	144
Afg	36	36	36	36	36
M/SME	43.73	53.66	39.58	21.43	20.00
ESPV	0.8	0.9	0.9	1.3	0.8

As seen, all but one of the ESPVs were below 1.0 in these examples. It is believed that, although the barrel temperature was allowed to vary in accordance with the invention, the moisture content in the barrel was too high to result in an acceptable product given the other conditions.

10

Thus, it is seen that the invention provides a satisfactory cold-water soluble starch. The starch may be prepared by extrusion in a conventional extruder.

While particular embodiments of the invention have been shown, it will be understood that the invention is not limited thereto since modifications may be made by those skilled in the art, particularly in light of the foregoing teachings. It is, therefore, contemplated by the appended claims to cover any such modifications as incorporate those features, which constitute the essential features of these improvements within the true spirit and scope of the invention. All references cited herein are hereby incorporated by reference.

15

20

WHAT IS CLAIMED IS:

1. An extruded starch product prepared by a process comprising:
providing a hydroxyalkyl starch, said starch being derivatized with a hydroxyalkyl substituent having from 2 to 6 carbon atoms; and
extruding said starch in an extruder, said extruder having a barrel, a die, and at least one rotating shaft, said barrel having at least first and second zones, said first zone being upstream from said second zone, the temperature in said first zone being insufficient to gelatinize said starch and the temperature in said second zone being sufficient to gelatinize said starch, said starch being extruded in the presence of total moisture in said barrel no greater than about 25% by weight of said starch, said process including the step of controlling the rotational speed of said shaft to impart a specific mechanical energy to said starch sufficient to result in a soluble extruded starch product that is capable of extrusion through said die at said rotational speed, said starch being substantially completely soluble in water at 25° C and being film-forming in aqueous solution.
2. A starch according to claim 1, said starch having a moisture content below about 15%.
3. A starch according to claim 1, said starch having a moisture content ranging from about 9% to about 12%.

ABSTRACT

Disclosed is a cold-water soluble starch and a process for preparing same. Generally, the process comprises providing a hydroxyalkyl starch and applying a shearing force to the starch in an extruder in the presence of moisture, the force and the moisture each being sufficient to gelatinize at least substantially all of the granules of the starch to thereby form a sheared starch. The starch is heated to its gelatinization temperature after the starch has passed partially through the barrel of the extruder, with the moisture being maintained at a level sufficiently high to allow gelatinization but sufficiency low to protect the starch from becoming too sticky to extrude. The extruded starch product thus formed may be used in connection with a number of film-forming, coating, and other applications.

	2A	2B	2C	2D	2E	2F	2G
Raw Material Information							
Substrate	B760	B760	B760	B760	B760	B760	B760
Dry Recipe % moisture	10-11	10-11	10-11	10-11	10-11	10-11	10-11
Feed Starch Rate (lbs/hr)	350	350	250	270	290	310	330
Cylinder Information							
Steam Flow to Cylinder lb/hr	12	12	12	12	12	12	12
Water Flow to Cylinder lb/hr	8	8	8	8	8	8	8
Extrusion Information							
Extruder Shaft Speed rpm	350	350	250	270	290	310	330
Extruder Motor Load %	51	51	35	40	41	46	47
Steam Flow to Extruder lb/hr	0	0	0	0	0	0	0
Water Flow to Extruder lb/hr	10	10	10	10	10	10	10
1 st Head Temp	98° F	97° F	96° F	96° F	95° F	95° F	97° F
2 nd Head Temp	135° F	136° F	124° F	125° F	125° F	126° F	126° F
3 rd Head Temp	144° F	198° F	129° F	131° F	132° F	135° F	136° F
4 th Head Temp	179° F	180° F	176° F	175° F	175° F	180° F	180° F
5 th Head Temp	292° F	295° F	231° F	233° F	233° F	236° F	239° F
Specific Mechanical Energy kW/ton	79	79	76	81	77	81	78

The starches produced were substantially completely soluble (over 99%).

Example 3

- The starch used in Example 1 was extruded on a Wenger TX57 Twin Screw
- 5 Extruder according to the following conditions and with the screw configuration shown in Figure 5.

Raw Material Information	
Substrate	B790
Dry Recipe % moisture	~11%
Dry Recipe Rate lb/hr	175
Feed Screw Speed rpm	15
Cylinder Information	
Cylinder Speed rpm	278
Steam Flow to Cylinder lb/hr	0
Water Flow to Cylinder lb/hr	0
Extrusion Information	
Extruder Shaft Speed rpm	398
Extruder Motor Load %	42
Steam Flow to Extruder lb/hr	0
Water Flow to Extruder lb/hr	11
Knife Speed rpm	459
No. of Knives	2
1 st Head Temp	77° F
2 nd Head Temp	78° F
3 rd Head Temp	108° F
4 th Head Temp	133° F
5 th Head Temp	270° F
6 th Head Temp	
Die Hole Size & How many?	3mm/15
Die Pressure psi	500
Vacuum on/off inches of vac?	OFF
Specific Mechanical Energy kW/ton	148

The expanded, friable product thus formed needed no drying. The product was ground on a Wiley Mill followed by an Alpine Mill to give a powder.

The powder was mixed into water at room temperature. A paste was formed, thus evidencing the gelatinized nature of the product. The paste was drawn into a thin film using a Meyer Road and then left to dry overnight at 50% relative humidity and 72° F to form a clear, transparent film.

Example 4

Cold Water Soluble Hydroxypropyl Starch

A cross-linked hydroxypropyl starch (B992 PURE-GEL[®], available from Grain Processing Corporation of Muscatine, Iowa), and having a moisture content of about 11% was extruded on a Wenger TX52 Twin Screw Extruder according to the conditions provided below and using the screw configuration shown in Figure 6.

	4A	4B	4C
Raw Material Information			
Substrate	B992	B992	B992
Dry Recipe % moisture	~11	~11	~11
Feed Screw Rate rpm	12	12	12
Cylinder Information			
Cylinder Speed rpm	110	110	110
Steam Flow to Cylinder lb/hr	0	0	0
Water Flow to Cylinder lb/hr	27.1	27.1	27.1
Extrusion Information			
Extruder Shaft Speed rpm	160	160	160
Extruder Motor Load %	29	28	17
Steam Flow to Extruder lb/hr	0	0	6.4
Water Flow to Extruder lb/hr	5.5	16.7	4.8
1 st Head Temp			
2 nd Head Temp	32° C	33° C	42° C
3 rd Head Temp	32° C	33° C	42° C
4 th Head Temp	90° C	90° C	90° C
5 th Head Temp	90° C	90° C	90° C
6 th Head Temp	65° C	65° C	65° C
7 th Head Temp	62° C	57° C	65° C
8 th Head Temp	62° C	57° C	65° C
9 th Head Temp	63° C	63° C	65° C
Die Pressure kPa	1720	70	2760

The extruded product, which was in the form of a condensed bead was dried on a moving grate dryer and then ground into a powder.

Each powder was mixed into water at room temperature to give pastes at 12% solids (thus evidencing the gelatinized nature of the extruded product). The pastes were
5 evaluated for gel strength and clarity. Gel strength was determined using a Texture Analyzer, Stevens LFRA Texture Analyzer TA 1000, 1 cm diameter probe after one day refrigeration at 40° F. Clarity was determined by observation on a scale of 0 to 9, 0 being opaque and 9 being clearest. The following results were obtained.

Evaluation Test	Water Temperature	4A	4B	4C	B992 Starch (control)
Gel Strength	30° C	26	25	26	*
	40° C	24	49	51	*
	50° C	50	56	60	48
	65° C	49	60	62	103
Clarity	30° C	1	1	1	*
	40° C	3	5	4	*
	50° C	7	7	6	0
	65° C	9	8	8	8

*The control, B992 Starch, was not amenable to testing at 30° C and 40° C.

Starch B992 was not amenable to testing at 30° and 40° because these temperatures were too low to allow this starch to gelatinize. Gel strength reflects the thickening power of a product when the product is mixed with water (generally, a higher gel strength is preferred in many applications).

5

Example 5

Cold Water Soluble Hydroxyethyl Starch

A hydroxyethyl starch (K95F COATMASTER® starch available from Grain Processing Corporation of Muscatine, Iowa) and having a moisture content of about 10 11% was extruded on a Wenger TX57 Twin Screw Extruder under the following conditions and having the screw configuration shown in Figure 7.

Raw Material Information	
Substrate	K95F
Dry Recipe % moisture	~11%
Dry Recipe Rate lb/hr	146
Feed Screw Speed rpm	8
Cylinder Information	
Cylinder Speed rpm	3498
Steam Flow to Cylinder lb/hr	0
Water Flow to Cylinder lb/hr	0
Extrusion Information	
Extruder Shaft Speed rpm	324
Extruder Motor Load %	32
Steam Flow to Extruder lb/hr	0
Water Flow to Extruder lb/hr	13
Knife Speed rpm	1087
No. of Knives	1
1 st Head Temp	85° F
2 nd Head Temp	169° F
3 rd Head Temp	175° F
4 th Head Temp	203° F
5 th Head Temp	245° F
6 th Head Temp	
Die Hole Size & How many?	3/36
Die Pressure psi	200
Vacuum on/off inches of vac?	OFF
Specific Mechanical Energy kW/ton	110

The product was made into an aqueous paste containing 35% extrudate at room temperature (thus evidencing the gelatinized nature of the product). The paste was

tested in a Rapid Visco-Analyzer (Newport Scientific) by monitoring the rotational viscosity and was found to have a viscosity of 1800 cP at 50° C compared to a baseline viscosity measurement of the raw starting material of about 100cP, thus evidencing the gelatinized nature of the extruded product. Additionally, when the paste was tested in a
5 Rapid Visco-Analyzer by monitoring the rotational viscosity during a controlled heating of the paste, the product exhibited no gelatinization peak. The starting material exhibited a crisp, characteristic peak at 70° C.

Example 6

10 Tack Coating and Cooked Products

The expanded starch from Example 1, 18 parts by weight, was blended with MALTRIN® M180 (a maltodextrin available from Grain Processing Corporation of Muscatine, Iowa), 9 parts by weight to form a dry blend. Water, 73 parts by weight, was added to a kettle and stirred with a powered mixer so as to create a vortex. The dry
15 blend was slowly added to the vortex, and the contents were mixed for an additional 10 to 30 minutes to form an instant tack coating.

The tack coating may be applied to a dry feed product substrate, such as a corn curl, pretzels, snack mix, or like item. The product may be applied by spraying or ladling at a level of from about 1% to about 15% weight gain, including moisture.
20 Seasonings, including savory seasonings such as Cajun, barbeque, cheese, mustard, ranch, Creole, and the like, or sweet seasonings such as sugar and pareils, may be added, and may be applied in any suitable manner, such as by hand or using a seasoning applicator. The resulting coated product preferably is dried in an oven at a temperature ranging from 300° F to 450° F to a moisture content of from about 3% to 5%.

25

Example 7

Oil-Based Instant Coating

Soybean oil, 50 parts by weight, was added to a vessel equipped with good agitation. The cold water soluble starch from Example 1, 7 parts by weight, was added
30 to the stirred oil and mixing was continued in order to achieve a smooth mixture.

Water, 42 parts by weight, and lecithin, 1 part by weight, were added as an emulsifier and mixing was continued for 10 to 15 minutes in order to achieve a smooth mixture.

The coated product may be applied to a food substrate as discussed in Example 6. Preferably, the coated product is dried in an oven at 300° F to 350° F with forced air
5 to a moisture content of from 3% to 5% in the finished product.

Example 8

Coated Peanut Products

A dusting mixture was prepared by dry-blending together the product of
10 Example 1, 50 parts by weight, and MALTRIN® M100 (a maltodextrin available from Grain Processing Corporation, Muscatine, Iowa), 50 parts by weight. Blanched, unroasted medium runner peanuts were placed in a 16" ribbed candy pan rotating at 20 to 25 RPM. A 50% sucrose solution was poured into the pan in an amount effective to just wet the nuts to give about a 2% weight gain. The dusting mixture was
15 then applied until the surfaces of the dusted nuts appeared dry, to thus give about a 5 to 6% weight gain. The dusted nuts were then tumbled an additional 2 to 3 minutes, during which time they wet back. An additional dusting with the dusting mixture was administered in order to achieve a dry appearance. The dry appearing, dusted nuts were then recoated with the sucrose solution, and the resulting rewetted nuts were
20 dusted to dryness again with the dusting mixture. This alternating procedure of wetting with the sucrose solution followed by dusting to dryness with the dusting mixture was repeated until a final dry appearing dusted nut resulted having a 75 to 100% weight gain as compared to the starting peanuts. The coated nuts were roasted in an oven at 300° F for 40 minutes with occasional stirring to assure uniformity of the
25 roast. The roasted coated nuts were cooled to room temperature and placed back into the ribbed pan rotating at 20 to 25 rpm. Subsequently, the instant tack coating formulation from Example 6 was sprayed onto the roasted coated nuts to provide approximately 0.5% weight gain in a rotating pan in order to create a slight tackiness. McCormick Barbecue Seasoning F76161, 6% to 8% weight gain was added, and the
30 coated nuts were tumbled until the seasoning was well distributed.

The resulting coated product was dried in an oven to a moisture level of from 3% to 5%.

Example 9

5

Trail Mix Coating and Product

A mixture was prepared by dry-blending together sugar, 25 parts by weight, the product of Example 1, 15 parts by weight, MALTRIN QD[®] M500 (a maltodextrin available from Grain Processing Corporation, Muscatine, Iowa), 5 parts by weight, and lecithin, 0.2 parts by weight. Water, 54.8 parts by weight, was added to a kettle and stirred with a powered mixer so as to create a vortex. The dry blend was slowly added into the water at the top edge of the vortex, and the contents were mixed for an additional 10 minutes to form an instant trail mix coating.

The resulting coating was sprayed onto a commercially purchased trail mix, by a spray gun system in a tumbler at a level of 5% to 15% weight gain. The resulting coated trail mix was dried in an oven at 150° F to a moisture content of 10 to 12%.

Example 10

Tablet Coating

A coating for a 3/8" round lactose/micro-crystalline cellulose placebo tablet was made. The coating had the following composition.

Formulation

Ingredients	Percentage by Weight
Product from Example 1	12.0%
Water	88.0%
	100.0

To prepare the coating, the starch was mixed into water with good agitation. A Vector HiCoater HC 100 coating pan with 2 spraying guns was used to apply the

coating onto the tablets to result in a 2% weight gain on the tablet. The coating pan was set at the following conditions.

Inlet temperature	60-65°C
Exhaust temperature	38-42°C
Pan speed	8 RPM
Process air flow	590 CFM
Spray air volume	125 atomize/50 pattern PSI
Spray rate	130-150 ml/min.

5 A plasticizer, such as glycerin, polyethylene glycols (PEG), propylene glycol (PG), oleic acid, triacetin, and the like can be used to improve the physical and mechanical properties of starch. Surfactants such as di-glycerides, tartaric acid esters of fatty acids, propyleneglyco mono and diesters of fatty acids, polysorbate 60, calcium or sodium stearyl-2-lactylate, lactic stearate, sodium stearyl fumarate, 10 succinylated monoglyceride, ethoxylated mono and diglycerides, and the like optionally may be used to provide hydrophilicity. Likewise, polymers of gums, cellulose derivatives, starch derivatives or hydrolysis products, and microorganism products, synthetic polymers such as polyvinyl alcohol, polyvinyl acetate, polyurethane, polystyrene, and polyvinylpyrrolidone, and so forth can be used to 15 improve the performance of the starch, for example, by increasing the flexibility and strength of the film coating.

Example 11

Film Coating

20 A coating for a 3/8" round lactose/micro-crystalline cellulose placebo was made. The coating had the following composition.

Formulation

Ingredients	Percentage by Weight
Product of Example 1	5.0%
Hydroxypropyl methyl cellulose	5.0%
Propylene glycol	1.0%
Polysorbate 80	0.5%
PURE-DENT [®] B815 corn starch NF*	0.5%
Titanium Dioxide	2.0%
Color	0.2%
Water	<u>85.8%</u>
	100.0

*Available from Grain Processing Corporation, Muscatine, Iowa.

To prepare the coating, the starch was mixed into water with good agitation. A Vector HiCoater HC 100 coating pan with 2 spraying guns was used to apply the starch to tablets to result in a 3% weight gain on the tablets. The coating pan was set at the following conditions:

Inlet temperature	65-70°C
Exhaust temperature	40-45°C
Pan speed	8 RPM
Process air flow	575-595 CFM
Spray air volume	125 atomize/50 pattern PSI
Spray rate	170-180 ml/min.

Example 12

The properties of the starch extrusion may be characterized in part by an Extruder Solubilization Point Value (ESPV), which may be calculated as follows.

$$\text{ESPV} = \frac{1.71 \times 10^6 \times (M + M_{ws}) \times D^4}{(T_h - T_l) (M (F_{ws} C_{ps} + F_{ww} C_{pw}) + M_{ws} C_{pw}) (F_{fww}^5 \times M \times A_{fg})}$$

wherein

M = mass flow rate of starch through extruder (kg/s)

M_{ws} = flow rate of water through extruder (kg/s)

D = diameter of extruder barrel (m)

T_h = highest head temperature in barrel (° C)

T_l = lowest head temperature in barrel (° C)

F_{ws} = weight fraction of starch in feed

F_{ww} = weight fraction of water in feed

F_{fww} = weight fraction of water in the barrel

A_{fg} = grams of starch from viscosity test*(g)

C_{ps} = specific heat capacity of starch (J/kg)

C_{pw} = specific heat capacity of water (4186 J/kg)

- 5 *From the method disclosed in "The Estimation of Starch Paste Fluidities."
W.R. Fetzer and L.C. Kirst, J. Cereal Chem., American Ass'n of Cereal Chemists,
Vol. 36, No. 2 (U.S., March, 1959).

10 Preferably, the ESPV is greater than or equal to 1.0. Following is a table of extrusion
conditions and ESPVs for the extruded starch of Examples 1, 2 and 3.

Inputs	Ex. 3	Ex. 1A	Ex. 1B	Ex. 1C	Ex. 1D	Ex. 1E	Ex. 1F	Ex. 1G
SME, kW/ton	148	100	106.2	103.4	100.7	102	99.3	98.2
M, lb/hr	175	4200	4275	4300	4400	4400	4500	4500
Mws, lb/hr	11	296	298	303	303	307	325	319
T _b , deg F	270	320	287	312	313	319	372	306
T _i , deg F	77	101	144	95	127	95	110	97
Fws	0.89	0.884	0.884	0.884	0.884	0.887	0.887	0.887
Fww	0.11	0.116	0.116	0.116	0.116	0.113	0.113	0.113
Ffww	0.163	0.174	0.174	0.174	0.173	0.171	0.173	0.172
Dia, mm	57	144	144	144	144	144	144	144
Afg	36	36	36	36	36	36	36	36
M/SME	1.18	42.00	40.25	41.59	43.69	43.14	45.32	45.62
ESPV	1.0	1.1	1.7	1.1	1.3	1.1	0.9	1.2

Inputs	Ex. 2A	Ex. 2B	Ex. 2C	Ex. 2D	Ex. 2E	Ex. 2F	Ex. 2G
SME, kW/lb	79	79	76	81	77	81	78
M, lbs/hr	350	350	250	270	290	310	330
Mws, lbs/hr	30	30	30	30	30	30	30
T _b , deg F	292	295	231	233	233	236	239
T _i , deg F	98	97	96	96	95	95	97
Fws	0.895	0.895	0.895	0.895	0.895	0.895	0.895
Fww	0.105	0.105	0.105	0.105	0.105	0.105	0.105
Ffww	0.176	0.176	0.201	0.195	0.189	0.184	0.180
Dia, mm	57	57	57	57	57	57	57
Afg	13	13	13	13	13	13	13
M/SME	4.43	4.43	3.29	3.33	3.77	3.83	4.23
ESPV	1.0	1.0	1.1	1.1	1.2	1.2	1.3

All of the ESPVs were above 1.0.

Comparative Example

Unacceptably sticky products were prepared by extruding B790 PURE-COTE[®] starch on a Wenger TX144 Twin Screw Extruder under the following conditions.

5

Inputs	C-1A	C-1B	C-1C	C-1D	C-1E
SME, kW/ton	86.9	76.4	103.6	140	150
M, lbs/hr	3800	4100	4100	3000	3000
Mws, lbs/hr	380	370	382	238	300
T _h , deg F	269	269	269	293	305
T _l , deg F	97	87	90	96	86
Fws	0.88	0.884	0.884	0.88	0.884
Fww	0.12	0.116	0.116	0.12	0.166
Ffww	0.200	0.189	0.191	0.185	0.196
Dia, mm	144	144	144	144	144
Afg	36	36	36	36	36
M/SME	43.73	53.66	39.58	21.43	20.00
ESPV	0.8	0.9	0.9	1.3	0.8

As seen, all but one of the ESPVs were below 1.0 in these examples. It is believed that, although the barrel temperature was allowed to vary in accordance with the invention, the moisture content in the barrel was too high to result in an acceptable product given the other conditions.

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Thus, it is seen that the invention provides a satisfactory cold-water soluble starch. The starch may be prepared by extrusion in a conventional extruder.

15

While particular embodiments of the invention have been shown, it will be understood that the invention is not limited thereto since modifications may be made by those skilled in the art, particularly in light of the foregoing teachings. It is, therefore, contemplated by the appended claims to cover any such modifications as incorporate those features, which constitute the essential features of these improvements within the true spirit and scope of the invention. All references cited herein are hereby incorporated by reference.

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1. An extruded starch product prepared by a process comprising:
providing a hydroxyalkyl starch, said starch being derivatized with a hydroxyalkyl substituent having from 2 to 6 carbon atoms; and
extruding said starch in an extruder, said extruder having a barrel, a die, and at least one rotating shaft, said barrel having at least first and second zones, said first zone being upstream from said second zone, the temperature in said first zone being insufficient to gelatinize said starch and the temperature in said second zone being sufficient to gelatinize said starch, said starch being extruded in the presence of total moisture in said barrel no greater than about 25% by weight of said starch, said process including the step of controlling the rotational speed of said shaft to impart a specific mechanical energy to said starch sufficient to result in a soluble extruded starch product that is capable of extrusion through said die at said rotational speed, said starch being substantially completely soluble in water at 25° C and being film-forming in aqueous solution.
2. A starch according to claim 1, said starch having a moisture content below about 15%.
3. A starch according to claim 1, said starch having a moisture content ranging from about 9% to about 12%.

ABSTRACT

Disclosed is a cold-water soluble starch and a process for preparing same. Generally, the process comprises providing a hydroxyalkyl starch and applying a shearing force to the starch in an extruder in the presence of moisture, the force and the moisture each being sufficient to gelatinize at least substantially all of the granules of the starch to thereby form a sheared starch. The starch is heated to its gelatinization temperature after the starch has passed partially through the barrel of the extruder, with the moisture being maintained at a level sufficiently high to allow gelatinization but sufficiency low to protect the starch from becoming too sticky to extrude. The extruded starch product thus formed may be used in connection with a number of film-forming, coating, and other applications.